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(54) Detergent compositions and process for preparing them.

(57) A process is provided for the preparation of a granular detergent composition having a bulk density of at least 650 g/l, which comprises treating a particulate starting material in a high speed mixer/densifier, characterised in that 0.1 to 50% by weight as calculated on the granular detergent composition of a liquid surfactant composition is mixed with the starting material during this treating process, said surfactant composition comprising

(a) a sodium or potassium salt of an alkyl sulphate in an amount from 5 to 40% by weight;
(b) an alkoxylated nonionic surfactant in an amount from 60 to 95% by weight,
(c) the balance being water in an amount from 0 to less than 20% by weight.

EP 0 544 365 A1

TECHNICAL FIELD

The present invention relates to a process for the preparation of a granular detergent composition having a high bulk density and good powder properties. More in particular, it relates to a process for the continuous preparation of such detergent compositions, especially those with high detergent activity. Moreover, it relates to a granular detergent composition obtainable by the process of the present invention.

BACKGROUND AND PRIOR ART

Recently there has been considerable interest within the detergents industry in the production of detergent powders having a relatively high bulk density, for example 600 g/l and above.

Generally speaking, there are two main types of processes by which detergent powders can be prepared. The first type of process involves spray-drying an aqueous detergent slurry in a spray-drying tower. In the second type of process, the various components are dry-mixed and optionally agglomerated with liquids, e.g. nonionics.

The most important factor which governs the bulk density of a detergent powder is the bulk density of the starting materials in the case of a dry-mixing process, or the chemical composition of the slurry in the case of a spray-drying process. Both factors can only be varied within a limited range.

Therefore, a substantial increase in bulk density can only be achieved by additional processing steps which lead to the densification of the detergent powder. There are several processes known in the art leading to such densification. Particular attention has thereby been paid to the densification of spray-dried powders by post-tower treatment.

In view of increased environmental concern, it is desirable to produce high density detergent powder containing alkyl sulphate as an active detergent component. The reason is that this type of active detergent material is readily biodegradable and therefore environmentally friendly.

EP-A-337,330 (Henkel) relates to a continuous process for obtaining high bulk density detergent powder containing a considerable amount of anionic and nonionic surfactant material, said process comprising treating spray-dried detergent material in a high-speed mixer under addition of nonionic material, whereby the mean residence time in the mixer is from 10-60 seconds. Alkyl sulphate is not mentioned in this document.

EP-A-265,203 (Unilever) discloses liquid surfactant compositions comprising an anionic and a nonionic surfactant. This patent document also discloses the use of these compositions which comprises spraying these compositions onto a solid particulate absorbent material. In this document alkyl sulphate is explicitly mentioned as a possible anionic surfactant which could effectively be applied in the surfactant composition which is sprayed onto the absorbent material.

The disadvantages of this route are the limited level of active detergent material which can be dosed in this way and the necessity that the particulate solid material to which the liquid surfactant compositions are added, is an absorbent material. Furthermore, at increased levels of active detergent material sticky detergent powder having deteriorated powder properties could easily be produced in this way.

It has also been proposed to make high active alkyl sulphate containing granules and to postdose these granules to an essentially anionic-free concentrated base powder. This method of producing high active alkyl sulphate containing detergent particles is, however, not attractive. The reason is that it involves the separate drying of a hydrous alkyl sulphate paste which requires considerable amounts of energy, or, alternatively, as described in EP-A-402,112 (P&G), the incorporation into said high active detergent particles of non-detergent active, less biodegradable additives such as ethoxylated nonionic surfactant material including at least 9 ethylene oxide groups.

It is an object of the present invention to provide a process for obtaining high bulk density granular detergent compositions having a bulk density of at least 650 g/l and a high active detergent content. It is also an object to provide an environmentally friendly, low energy process for the preparation of a high bulk density granular detergent composition having a high active detergent content. It is a further object to provide a process for obtaining a detergent composition comprising alkyl sulphate as one of the active detergent constituents.

We have now found that these and other objects can be achieved if a liquid surfactant system comprising alkyl sulphate and an alkoxylated nonionic surfactant is thoroughly mixed with particulate starting material during treatment of this starting material in a high speed mixer/densifier.

DEFINITION OF THE INVENTION

In a first aspect, the present invention provides a process for the preparation of a granular detergent composition having a bulk density of at least 650 g/l, which comprises treating a particulate starting material in a high speed mixer/densifier, characterised in that 0.1 to 50% by weight as calculated on the granular detergent composition of a liquid surfactant composition is mixed with the starting material during this treating process, said surfactant composition comprising

(a) a sodium or potassium salt of an alkyl sulphate in an amount from 5 to 60% by weight;

(b) an alkoxylated nonionic surfactant in an amount from 40 to 95% by weight,

(c) the balance being water in an amount from 0 to less than 20% by weight.

In a second aspect, the invention provides a granular detergent composition obtainable by this process and having a particle porosity of less than 10%, preferably less than 5%.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is concerned with a process for the preparation of a high bulk density powder having a high active detergent content. An important characteristic of the present process is that the detergent material remains throughout the process in particulate or granular form. Caking, balling an dough formation are avoided and the final product does not require an additional step in which the particle size is reduced.

During the process of the invention, a liquid surfactant composition comprising alkyl sulphate and an alkoxylated,preferably ethoxylated, nonionic surfactant is thoroughly mixed with a particulate starting material in a high-speed mixer/densifier. This is essentially an agglomeration process, wherein the particulate starting material is agglomerated by the liquid surfactant material, resulting in detergent particles containing the particulate starting material and a surfactant phase. Generally, this surfactant phase acts as a binder for the particulate starting material.

The advantage of this agglomeration process over a process wherein the liquid surfactant composition is absorbed into the particulate starting material is the fact that by agglomerating much higher levels of liquid surfactant material can be incorporated in the detergent powder to be obtained, while maintaining good powder properties.

This agglomeration process can be carried out either as a continuous or as a batch process. For economic reasons, it is preferred to carry out the process of the invention continuously in a high-speed mixer/densifier, whereby the mean residence time is from about 5-30 seconds.

Furthermore, it is important that the agglomeration process is a well controlled, robust process resulting in detergent powder with the desired particle size and with powder properties which are comparable to those of detergent powders currently on the market . For obtaining detergent powder with good powder properties, it has been found effective to add to the liquid surfactant composition one or more components with such a composition that a significant viscosity increase of the resulting total liquid composition is obtained. The addition of these components raises the afore-mentioned viscosity generally by at least a factor 5, preferably by at least a factor 10, a viscosity increase by at least a factor 100 being most preferred (when measured in a Haake viscometer at a shear rate between 0.1 and 20 S^{-1}). As a result of this viscosity increase, the agglomeration process appeared to be better controllable resulting in better powder properties of the detergent material produced in this way.

Examples of such viscosity raising components are water and, particularly, fatty acid in combination with a stoichiometric amount of alkaline material (such as caustic soda) sufficient to neutralize the fatty acid which obviously results in the formation of soap.

For obtaining a very high bulk density powder, the detergent powder obtained by the process of the invention may be further treated in a second step in a moderate speed granulator/densifier, whereby it is brought into or maintained in a deformable state, the mean residence time being from 1-10 minutes, and thereafter in a third step in a drying and/or cooling apparatus, as described in EP-A-367,339.

Particulate starting material

The process of the present invention is very flexible with respect to the chemical composition of the particulate starting material. This material comprises the compounds usually found in detergent compositions such as builders and detergent active materials. Phosphate containing as well as zeolite containing compositions and compositions having either high or low active detergent content may be used as particulate starting material.

The detergency builder present in the starting material may be any material capable of reducing the level of free calcium ions in the wash liquor and will preferably provide the composition with other beneficial properties such as the generation of an alkaline Ph, the suspension of soil removed from the fabric and the suspension of fabric softening clay material. Examples of suitable builders include precipitating builders such as the alkali metal carbonates, bicarbonates, orthophosphates, sequestering builders such as the alkali metal tripolyphosphates or nitrilotriacetates, or ion exchange builders such as the amorphous alkali metal alumino-silicates or the zeolites.

The process is also suitable for producing calcite/sodium carbonate built detergent compositions.

Preferably, the builder material applied in the process of the present invention consists of fine particles, desirably with a particle size of less than 10 microns. When very high bulk density detergent powder is prepared, part of the builder material amounting to about 0.5-10% by weight as calculated on the total granular composition is preferably added during the second step when the detergent powder is further treated in a moderate speed granulator/densifier, as mentioned above. This process is disclosed in more detail by EP-A-390,251.

The level of builder material present in the starting material is preferably such that its content as calculated on the total granular composition is in the range from 10 to 70% by weight, most preferably from 30 to 60% by weight.

The detergent active material present in the starting material may be selected from anionic, ampholytic, zwitterionic or nonionic detergent active materials or mixtures thereof. Examples of suitable synthetic anionic detergent compounds are sodium and potassium (C_9-C_{20}) benzene sulphonates, particularly sodium linear secondary alkyl (C_{10}-C_{15}) benzene sulphonates; and sodium alkyl glyceryl ether sulphates, especially those ethers of the higher alcohols derived from tallow or coconut oil and synthetic alcohols derived from petroleum. Suitable nonionics which may be used as constituents of the particulate starting material include, in particular the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example, aliphatic alcohols, acids, amides or alkyl phenols with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Specific nonionic detergent compounds are alkyl (C_6-C_{22}) phenol ethylene oxide condensates, generally having 5 to 25 EO, i.e. 5 to 25 units of ethylene oxide per molecule, and the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, generally 5 to 40 EO. The level of detergent active material present in the starting material may be in the range from 0 to 30% by weight. This level is preferably less than 10% by weight, more preferably less than 5% by weight.

Other examples of materials which may be present in the particulate starting material include fluorescers; polycarboxylate polymers; antiredeposition agents, such as carboxy methyl cellulose; fatty acids; fillers, such as sodium sulphate; clays such as kaolin or bentonite.

The particulate starting material for the process of the invention may be prepared by any suitable method, such as spray-drying or dry-mixing. The components of the starting material may also effectively be added separately to the mixer/densifier. It is considered to be one of the advantages of the process of this invention that high bulk density, high active detergent powders may be prepared from dry-mixed or untreated starting materials, without the need for expensive spray-drying equipment. On the other hand, it may also be desirable that one or more of the ingredients of the starting material are adjuncts of liquids onto solid components, prepared by spray-drying, granulation or via insitu neutralization in a high-speed mixer.

The liquid surfactant system

The liquid surfactant composition which is mixed into the particulate starting material in the mixer/densifier comprises an anionic surfactant (which is a sodium or potassium salt of an alkyl sulphate), an alkoxylated nonionic surfactant and water. The amount of the liquid surfactant composition which is applied is such that its content as calculated on the total granular detergent obtained is in the range from 0.1 to 50% by weight, preferably from 20 to 50 % by weight, more preferably from 25 to 50 % by weight. Preferred surfactant compositions according to the invention contain not more than 30% by weight of alkyl sulphate, and as little water as possible. Compositions in which the weight ratio of alkyl sulphate to alkoxylated nonionic surfactant ranges from 0.125:1 to 0.5:1 are of especial interest.

The nonionic surfactant is preferably an ethoxylated or mixed ethoxy-propoxylated primary or secondary aliphatic alcohol. Most preferred are ethoxylated primary alcohols, especially C_8-C_{15} primary alcohols ethoxylated with from 2 to 25 moles of ethylene oxide per mole of alcohol. The anionic surfactant component of the liquid surfactant composition is a sodium or potassium alkyl sulphate salt. Suitable alkyl sulphates are sodium C_{12}-C_{18} alkyl sulphates, especially the primary alkyl sulphates, although other alkyl

sulphates outside this carbon chain length range, and potassium alkyl sulphates may also be used.

As described above, it is preferred to add to the liquid surfactant composition one or more components with such a composition, that a significant viscosity increase of the resulting total liquid composition is obtained. The total level of these components may be as high as 20% by weight as calculated on the total liquid composition, said level being preferably in a range of from 2 to 10% by weight.

The densification process and the final densified powder

It was found to be essential for obtaining an optimal densification to subject the particulate starting material to a three-step densification process, as extensively disclosed in EP-A-367,339.

The densified powder thus obtained has preferably a particle porosity of less than 10%, more preferably less than 5%. This powder may be used as a detergent powder in its own right. Generally, however, various additional ingredients may be added to give a more efficient product. The amount of post-dosed material will generally range from about 10 to 200% by weight, calculated on the weight of the densified powder.

Examples of materials which may be postdosed to the densified powder include enzymes, bleaches, bleach precursors, bleach stabilizers, lather suppressors, perfumes and dyes. Liquid or pasty ingredients may conveniently be absorbed on to solid porous particles, generally inorganic, which may then be postdosed to the densified powder obtained by the process of the invention.

The process of the invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise indicated. In the Examples the following abbreviations are used:

PAS :	Primary alkyl sulphate, sodium salt of C_{12}-C_{18} primary alkyl sulphate
NI :	C_{12}-C_{14} Nonionic surfactant (ethoxylated alcohol containing on average 5 EO groups, ex Kolb
Carbonate :	Sodium carbonate, ex AKZO
Silicate :	Sodium alkaline silicate
Zeolite :	Zeolite A4 (Wessalith [trade mark]),ex Degussa.
Soap :	Sodium salt of C_{16}-C_{22} fatty acid, ex Unichema
Polymer :	Sokalan CP5/7 [trademark) type of polymer, ex BASF
Sulphate :	Sodium sulphate.

COMPARATIVE EXAMPLES A,B

The following zeolite-containing detergent granules were prepared by spray-drying aqueous slurries. The compositions (in % by weight) of the porous granules thus obtained are shown in Table 1.

TABLE 1

Examples	A	B
Zeolite 4A	76.7	-
Sulphate	8.3	71.2
Carbonate	-	25.4
Polymer	-	1.7
Moisture	15.0	1.7
	100.0	100.0

The granules were free flowing and had a mean particle size of ca. 300 microns.

The granules were fed directly into a continuous low speed mixer, The rotational speed was in both cases about 30 rpm. The mean residence time of the granules in the mixer was approximately 2 minutes. In this apparatus, a mixture of 20 wt% PAS and 80 wt% non-ionic was sprayed onto these granules until the granules were almost saturated. At this stage, the free-flowiness of the granular detergent material started to decline. The following compositions and physical properties of the resulting detergent granules were obtained:

TABLE 2

Examples	A	B
Compositions:		
Zeolite 4A	57.6	-
Sulphate	6.2	53.4
Carbonate	-	19.0
Polymer	-	1.3
Moisture	11.2	1.3
PAS	5.0	5.0
Nonionic 3EO	20.0	20.0
	100.0	100.0
Physical properties:		
Bulk density (g/l)	830	690
Dynamic Flow Rate (ml/s)	85	103
Particle size (microns)	320	270

It can be seen that the maximum level of detergent active material which can be sprayed-on in view of the obtainable powder properties, is 25 % by weight. Furthermore, it can be derived by comparing the particle size of the detergent granules before and after treatment in the mixer, that no agglomeration has occurred.

EXAMPLE 1

Several detergent components of which the solid components have a particle size lower than 200 microns, were fed into a high speed batch mixer/densifier. The mean residence time of the granular detergent mixture in the batch mixer/densifier was approximately 3 minutes.
The composition of the granular detergent powder leaving the batch mixer/densifier is given in Table 3.

TABLE 3

Example	1
Zeolite 4A	43.7
Carbonate	16.2
PAS	8.3
Nonionic	19.5
Water	12.3
	100.0

The thus obtained granular detergent compositions had good powder properties (DFR was 101 ml/s) and a bulk density of about 770 g/l .
It can be seen that the level of the active detergent material present in the detergent powder obtained (i.e.: 27.8 % by weight) is higher than the levels obtained in the comparative examples.

EXAMPLES 2,3

Several detergent components of which the solid materials have a particle size lower than 200 microns, were fed into a Lödige (Trade Mark) Recycler CB30, a continuous high speed mixer/densifier. The rotational speed was 1600 rpm. The mean residence time of the granular mixtures in the Lödige Recycler was

approximately 10 seconds.

The compositions of the granular material leaving the Lödige Recycler are given in Table 4.

TABLE 4

Examples Compositions:	2	3
Zeolite 4A	52.6	47.1
Carbonate	-	8.0
PAS	8.5	8.3
NI	19.4	18.8
Soap	2.9	2.9
Water	16.4	14.9
	100.0	100.0

The thus obtained granular detergent compositions had good powder properties, a bulk density of about 700 g/l and a particle size of 500-600 microns.

It can be seen that the levels of the active detergent material present in the detergent powders obtained are respectively 30.8 % by weight and 30.0 % by weight. These active detergent levels are much higher than the levels obtained in the comparative examples and also higher than the active detergent level obtained in example 2. This is the result of incorporating into the liquid surfactant composition fed into the Recycler, fatty acid in combination with a stoichiometric amount of caustic soda, as viscosity raising material. It is clear that during the mixing/densifying process soap is formed from this material.

Claims

1. Process for the preparation of a granular detergent composition having a bulk density of at least 650 g/l, which comprises treating a particulate starting material in a high speed mixer/densifier, characterised in that 0.1 to 50% by weight as calculated on the granular detergent composition of a liquid surfactant composition is mixed with the starting material during this treating process, said surfactant composition comprising
   (a) a sodium or potassium salt of an alkyl sulphate in an amount from 5 to 60% by weight;
   (b) an alkoxylated nonionic surfactant in an amount from 40 to 95% by weight,
   (c) the balance being water in an amount from 0 to less than 20% by weight.

2. Process according to claim 1, wherein the process is carried out continuously, whereby the mean residence time in the high speed mixer/densifier is from 5-30 seconds.

3. Process according to claim 1 or claim 2, wherein the level of the liquid surfactant system mixed with the starting material is in the range of from 20 to 50% by weight, preferably from 25 to 50% by weight.

4. Process according to any of claims 1-3, wherein in addition to the liquid surfactant composition one or more components are mixed with the starting material, said components being of such composition that the viscosity of the resulting total liquid composition is increased.

5. Process according to claim 4, wherein said additional components contain fatty acid in combination with a stoichiometric amount of alkaline material sufficient to neutralize the fatty acid.

6. Process according to claim 4 or 5, wherein the total level of said additional components is at most 20% by weight, preferably in the range of from 2 to 10% by weight, as calculated on the resulting total liquid composition.

7. Process according to any of claims 1-6, wherein the particulate starting material is further treated
   (i) in a second step in a moderate speed granulator/densifier, whereby it is brought into, or maintained in, a deformable state, the mean residence time being from about 1-10 minutes and
   (i) in a third step in a drying and/or cooling apparatus.

8. Process according to any of claims 1-7, wherein the component (a) present in the liquid surfactant composition is a sodium salt of a C_{12}-C_{18} primary alkyl sulphate.

9. Process according to any of claims 1-8, wherein the component (b) present in the liquid surfactant composition is an ethoxylated nonionic surfactant of the formula $R(OC_2H_4)_nOH$, whereby R is a C_8-C_{15} alkyl group and n ranges from 2 to 25.

10. Granular detergent composition obtainable by the process according to any of claims 1-10 and having a particle porosity of less than 10%, preferably less than 5%.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 92 20 3563

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	EP-A-0 451 894 (UNILEVER NV ET. AL.) * page 3, line 15 - page 5, line 25 * ---	1-10	C11D17/06 C11D1/83 C11D11/00
A	EP-A-0 420 317 (UNILEVER NV ET. AL.) * the whole document * ---	1-10	
A	EP-A-0 425 277 (UNILEVER PLC ET. AL.) * page 2, line 35 - page 8, line 20 * ---	1-5,7,9,10	
A	EP-A-0 390 251 (UNILEVER NV ET. AL.) * page 3, line 30 - page 6, line 25; examples 1-3 * -----	1,2,7,10	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C11D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	03 MARCH 1993	DOOLAN G.J.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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